


S

ENGINEERING CHANGE NOTICE

Page 1 of 7

1 ECN 656221

Proj
ECN N/A

2 ECN Category (mark one) Supplemental <input type="radio"/> Direct Revision <input checked="" type="radio"/> Change ECN <input type="radio"/> Temporary <input type="radio"/> Standby <input type="radio"/> Supersedure <input type="radio"/> Cancel/Void <input type="radio"/>	3 Originator's Name Organization MSIN and Telephone No J A Compton 1AF00 T5-12 373-2296		4 USQ Required? <input checked="" type="radio"/> Yes <input type="radio"/> No	5 Date 01/27/00
	6 Project Title/No /Work Order No Prototype Vertical Denitration Calciner / 110890	7 Bldg /Sys /Fac No 234-5Z / 86B	8 Approval Designator N/A	
	9 Document Numbers Changed by this ECN (includes sheet no and rev) HNF-SD-CP-TP-088, Rev 10	10 Related ECN No(s) N/A	11 Related PO No N/A	
12a Modification Work <input type="radio"/> Yes (fill out Blk 12b) <input checked="" type="radio"/> No (NA Blks 12b 12c, 12d)	12b Work Package No N/A	12c Modification Work Completed N/A Design Authority/Cog Engineer Signature & Date	12d Restored to Original Condition (Temp or Standby ECNs only) N/A Design Authority/Cog Engineer Signature & Date	
13a Description of Change 1 Changed text in Procedure section to indicate that a vacuum controller has been replaced by a globe valve 2 Changed text in Procedure section to have workers contact the 241-Z Cognizant Engineer with requests for spent scrub solution disposals, rather than contacting the PFP Environmental Team 13b Design Baseline Document? <input type="radio"/> Yes <input checked="" type="radio"/> No				
14a Justification (mark one) Criteria Change <input type="radio"/> Design Improvement <input checked="" type="radio"/> Environmental <input type="radio"/> Facility Deactivation <input type="radio"/> As Found <input type="radio"/> Facilitate Const <input type="radio"/> Const Error/Omission <input type="radio"/> Design Error/Omission <input type="radio"/>	14b Justification Details Change 1 needed for replacing a failed vacuum controller Vacuum controller was always fully open, anyway, and, thus, did no controlling Manual globe valve will perform the same function, be less prone to failure, and occupy less glovebox space Change 2 needed to correct the personnel to contact for spent scrub solution disposal Correct person/organization was still being determined due to PFP Redesign when Revision 10 was issued			
15 Distribution (include name MSIN and no of copies) See Distribution Sheet			RELEASE STAMP <div style="border: 2px solid black; padding: 10px; text-align: center;"> <div style="display: flex; justify-content: space-between;"> <div> JAN 28 2000 DATE STA. 5 </div> <div>  </div> <div> <div style="border: 1px solid black; border-radius: 50%; width: 40px; height: 40px; display: flex; align-items: center; justify-content: center;">12</div> ID </div> </div> </div>	

ENGINEERING CHANGE NOTICE

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1 ECN (use no from pg 1)

656221

16 Design Verification Required

☐ Yes

☒ No

17 Cost Impact

ENGINEERING

Additional ☐ \$ N/A

Savings ☐ \$ N/A

CONSTRUCTION

Additional ☐ \$ N/A

Savings ☐ \$ N/A

18 Schedule Impact (days)

Improvement ☐ N/A

Delay ☐ N/A

19 Change Impact Review Indicate the related documents (other than the engineering documents identified on Side 1) that will be affected by the change described in Block 13 Enter the affected document number in Block 20

SDD/DD	<input type="checkbox"/>	Seismic/Stress Analysis	<input type="checkbox"/>	Tank Calibration Manual	<input type="checkbox"/>
Functional Design Criteria	<input type="checkbox"/>	Stress/Design Report	<input type="checkbox"/>	Health Physics Procedure	<input type="checkbox"/>
Operating Specification	<input type="checkbox"/>	Interface Control Drawing	<input type="checkbox"/>	Spares Multiple Unit Listing	<input type="checkbox"/>
Criticality Specification	<input type="checkbox"/>	Calibration Procedure	<input type="checkbox"/>	Test Procedures/Specification	<input type="checkbox"/>
Conceptual Design Report	<input type="checkbox"/>	Installation Procedure	<input type="checkbox"/>	Component Index	<input type="checkbox"/>
Equipment Spec	<input type="checkbox"/>	Maintenance Procedure	<input type="checkbox"/>	ASME Coded Item	<input type="checkbox"/>
Const Spec	<input type="checkbox"/>	Engineering Procedure	<input type="checkbox"/>	Human Factor Consideration	<input type="checkbox"/>
Procurement Spec	<input type="checkbox"/>	Operating Instruction	<input type="checkbox"/>	Computer Software	<input type="checkbox"/>
Vendor Information	<input type="checkbox"/>	Operating Procedure	<input type="checkbox"/>	Electric Circuit Schedule	<input type="checkbox"/>
OM Manual	<input type="checkbox"/>	Operational Safety Requirement	<input type="checkbox"/>	ICRS Procedure	<input type="checkbox"/>
FSAR/SAR	<input type="checkbox"/>	IEFD Drawing	<input type="checkbox"/>	Process Control Manual/Plan	<input type="checkbox"/>
Safety Equipment List	<input type="checkbox"/>	Cell Arrangement Drawing	<input type="checkbox"/>	Process Flow Chart	<input type="checkbox"/>
Radiation Work Permit	<input type="checkbox"/>	Essential Material Specification	<input type="checkbox"/>	Purchase Requisition	<input type="checkbox"/>
Environmental Impact Statement	<input type="checkbox"/>	Fac Proc Samp Schedule	<input type="checkbox"/>	Tickler File	<input type="checkbox"/>
Environmental Report	<input type="checkbox"/>	Inspection Plan	<input type="checkbox"/>		<input type="checkbox"/>
Environmental Permit	<input type="checkbox"/>	Inventory Adjustment Request	<input type="checkbox"/>		<input type="checkbox"/>

20 Other Affected Documents (NOTE Documents listed below will not be revised by this ECN) Signatures below indicate that the signing organization has been notified of other affected documents listed below

Document Number/Revision

Document Number/Revision

Document Number/Revision

21 Approvals

Signature	Date	Signature	Date
Design Authority JA Compton <i>James A. Compton</i>	1-27-00	Design Agent	
Cog Eng JA Compton <i>James A. Compton</i>	1-27-00	PE	
Cog Mgr CS Sutter <i>Caroline Sutter</i>	1/27/00	QA	
QA		Safety	
Safety		Design	
Environ		Environ	
Other		Other	

DEPARTMENT OF ENERGY

Signature or a Control Number that tracks the Approval Signature

ADDITIONAL

UNREVIEWED SAFETY QUESTION (USQ)

ECN 655246 SCREENING AND EVALUATION

1	Identification Number	22-00-00104 ^{gac} 01-19-00	USQ SCREENING	Page 1 of 3
2	Title	REPLACE CALCINER VACUUM CONTROLLER WITH GLOBE VALVE		

INSTRUCTIONS Respond to each question and provide justification for each response. A restatement of the question does not constitute a satisfactory justification or basis. An adequate justification provides sufficient explanation such that an independent reviewer could reach the same conclusion based on the information provided [DOE 5480 21, 10 e 1]

DESCRIPTION

The vacuum controller used in the prototype vertical calciner will be replaced with a manual globe valve per Work Package ~~22-00-00104~~ and Engineering Change Notice 655246. The globe valve will work as well as the vacuum controller, which was always fully open during calciner testing. The valve will also have fewer methods of failure and will be less of an obstruction inside the glovebox.

~~22-99-0319~~ ^{gac} ~~01-19-00~~ ~~22-00-00104~~ ^{gac} ~~01-27-00~~

INTRODUCTION

The vacuum controller in the off gas line for the prototype vertical calciner failed closed while starting the process for a test run. This failure ended the gas flow through the off-gas system and aborted that attempt to run the calciner. A manual globe valve was chosen to replace the vacuum controller for the following reasons:

- The vacuum controller has been adjusted to fully open since the first few tests due to the need to maintain the calciner interior pressure as low as possible. Lowering the internal pressure allows more capacity for gas formation from solution boiling and chemical decomposition while keeping the internal pressure below that of the surrounding atmosphere. Because the controller was fully open all the time, it performed no automatic control function, which was proven by the observation of fluctuations in the calciner pressure indications on gauges as each run progressed through start-up, feeding and shut-down. The manual globe valve will work identically to what the vacuum controller was achieving.

- Temporary restrictions on the calciner glovebox air lock usage made replacement by an available identical vacuum controller impossible in a timely fashion.

- The globe valve is a smaller obstruction to working inside the glovebox gloves. Elimination of the sensing line between the calciner and vacuum controller removes another obstruction.

AFFECTED SS&Cs

The only systems, structures, and components affected by this change is Glovebox 188-1 in the Plutonium Process Support Laboratories and the calciner system contained within that glovebox. This glovebox is located in Room 188 of the 234-5Z Building in the Plutonium Finishing Plant Complex.

AUTHORIZATION BASIS

- 1 HNF-SD CP-SAR-021, Revision 1, *Plutonium Finishing Plant Final Safety Analysis Report*, Sections 6 5 1 1 1 'Plutonium Process Support Laboratories,' 6 5 4 2, "Major Components," and 9 1 A, "Abnormal Operations for the Plutonium Bearing Solution Stabilization Process."
- 2 WHC SD-CP-SAR-021, Revision 0-L, Addendum to WHC-SD CP-SAR-021, Sections 2 3 3 2 7, "26-in -Hg Vacuum System," and 3 2 2 "Abnormal Occurrence Selection."
- 3 WHC-SD-CP-OSR 010, Revision 0 K, *Operational Safety Requirements for the Plutonium Finishing Plant*.

No changes are required or proposed to these Authorization Basis documents.

UNREVIEWED SAFETY QUESTION (USQ)

ECN 655246 SCREENING AND EVALUATION

1	Identification Number	22-00-00104-19-00	USQ SCREENING	Page 2 of 3
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2	Title	REPLACE CALCINER VACUUM CONTROLLER WITH GLOBE VALVE
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CONCLUSION

The proposed work is bounded by the accident descriptions contained in the Plutonium Finishing Plant Authorization Basis. One question within the Unreviewed Safety Question Screening section is answered "Yes" because of a change to the facility as described in the Authorization Basis. An Unreviewed Safety Question Evaluation is required. The questions in the associated Unreviewed Safety Question Evaluation are all answered "No," therefore, this proposed change does not present an Unreviewed Safety Question.

REFERENCES

None

QUESTIONS

- 1 Does the proposed change or occurrence represent a change to the facility or procedures as described in the Authorization Basis?

☐ N/A ☐ No ☒ Yes/Maybe

BASIS Yes. The vacuum controller is mentioned specifically in both Revision 1 (Section 6.5.4.2, "Major Components,") and the Addendum to Revision 0-L (Section 2.3.3.2.7, "26-in -Hg Vacuum System") of the Authorization Basis. Replacement of the vacuum controller with a manual globe valve will not change any functionality of the off-gas system because the vacuum controller operated in a constantly fully open position and, hence, exercised no controlling function. The globe valve will usually be left in a constant position once the proper position is determined during tests. Occasional adjustments may be made due to changing processing conditions between runs, just as the vacuum controller would have reacted to such changes. Replacement of the vacuum controller with a globe valve is a change to the facility as described in the Authorization Basis despite the lack of change in operating functionality.

No changes to the standard facility procedures are needed or requested in order to accomplish this work.

- 2 Does the proposed change or occurrence represent conditions that have not been analyzed in the Authorization Basis?

☐ N/A ☒ No ☐ Yes/Maybe

BASIS Neither HNF-SD-CP-SAR 021, Revision 1, *Plutonium Finishing Plant Final Safety Analysis Report*, Section 9.1A, "Abnormal Operations for the Plutonium-Bearing Solution Stabilization Process" nor WHC-SD-CP-SAR 021, Revision 0 L, Addendum to WHC-SD-CP-SAR-021, Section 3.2.2, "Abnormal Occurrence Selection," mention credible accident scenarios related to changing control of the calciner vacuum level or the method of achieving that control. Because the globe valve will function identically to the vacuum controller, there will be no change to the actual operating conditions with the vertical calciner system. All aspects of this work and potential consequences are bounded by the safety systems and accident analyses described in the Authorization Basis.

UNREVIEWED SAFETY QUESTION (USQ)

ECN 65524-6 SCREENING AND EVALUATION

1	Identification Number	22-00-00104 ^{00C} 1/19-00	USQ SCREENING	Page 3 of 3
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2	Title	REPLACE CALCINER VACUUM CONTROLLER WITH GLOBE VALVE
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- 3 Does the proposed change represent a test or experiment NOT described in the Authorization Basis that may affect the safe operation of the facility?
☐ N/A ☒ No ☐ Yes/Maybe

BASIS The proposed work is neither a test nor an experiment

- 4 Does the proposed change or occurrence represent a change to the Technical Safety Requirements or a reduction in the margin of safety defined in the Technical Safety Requirements?
☒ N/A ☐ No ☐ Yes/Maybe

BASIS There are no Technical Safety Requirements or Operational Safety Requirements in WHC-SD-CP-OSR 010, Revision 0-K, *Operational Safety Requirements for the Plutonium Finishing Plant*, regarding control of the calciner vacuum level. There are no margins of safety for PFP's OSRs. No changes to the existing Operational/Technical Safety Requirements are requested for this work.

USQE #1 JA Compton

USQE #2 JP King

(Print Name)

(Print Name)

James A. Compton
Signature

Date 1-13-00

Jeffrey P. King
Signature

Date 1/14/00

If there is a YES/MAYBE response to questions 1, 2, 3, or 4, then a USQ Evaluation must be completed.

The following guidance should be considered when completing this screening. This guidance should not be considered all inclusive. Additional factors may need to be considered depending on the nature of the proposed change.

Does the proposed change

- 1) Modify, add, or delete a safety class function of a structure, system, or component stated in the authorization basis?
- 2) Alter the design of a structure, system, or component as described in the authorization basis?
- 3) Modify, add, or delete the description of operation, operating environment, or analyses of any system or component described in the authorization basis?
- 4) Modify, add, delete, or conflict with any of the design bases stated in the authorization basis?
- 5) Conflict with the principle or general design criteria stated in the authorization basis?
- 6) Modify, add, or delete any plant design features described in the authorization basis?
- 7) Modify, add, or delete a flow diagram or facility drawing provided in the authorization basis?
- 8) Create the potential for new system or component interactions (e.g., seismic, electrical, breaker coordination)?

UNREVIEWED SAFETY QUESTION (USQ)

Identification Number

22-00-00104 ECN 655246

USQ EVALUATION

Page 1 of 2

Title REPLACE CALCINER VACUUM CONTROLLER WITH GLOBE VALVE

- 1 Does the PROPOSED ACTIVITY or DISCOVERY increase the probability of occurrence of an accident previously evaluated in the AUTHORIZATION BASIS documentation?

X No ☐ Yes/Maybe

Basis The proposed change is unrelated to any of the occurrences or accidents postulated in the Authorization Basis

- 2 Does the PROPOSED ACTIVITY or DISCOVERY increase the consequences of an accident previously evaluated in the AUTHORIZATION BASIS documentation?

X No ☐ Yes/Maybe

Basis The proposed change is unrelated to any of the occurrences or accidents postulated in the Authorization Basis

- 3 Does the PROPOSED ACTIVITY or DISCOVERY increase the probability of occurrence of a malfunction of EQUIPMENT IMPORTANT TO SAFETY (ITS EQUIPMENT) previously evaluated in the AUTHORIZATION BASIS documentation?

X No ☐ Yes/Maybe

Basis The proposed change is unrelated to any of the Equipment Important To Safety listed in the Authorization Basis The Equipment Important To Safety in this case is the glovebox enclosure Failure to maintain control of the vacuum level in the calciner with the vacuum controller or the globe valve does not affect the probability that the glovebox will contain any release of powder from inside the calciner

- 4 Does the PROPOSED ACTIVITY or DISCOVERY increase the consequences of a malfunction of ITS EQUIPMENT previously evaluated in the AUTHORIZATION BASIS documentation?

X No ☐ Yes/Maybe

Basis The proposed change can not affect any of the Equipment Important To Safety listed in the Authorization Basis or the consequences of failure The consequences are related to the integrity of the glovebox, which is unrelated to the presence of the vacuum controller or the globe valve

- 5 Does the PROPOSED ACTIVITY or DISCOVERY create the possibility of an accident of a different type than any previously evaluated in the AUTHORIZATION BASIS documentation?

X No ☐ Yes/Maybe

Basis The proposed manual globe valve will function in the same way as the existing vacuum controller There were no postulated accidents or abnormal occurrences for the vertical calciner regarding the vacuum controller in the accident analyses of the Authorization Basis

UNREVIEWED SAFETY QUESTION (USQ)

Identification Number

22-00-00104 ECN 655246

USQ EVALUATION

Page 2 of 2

900 1-17-00

- 6 Does the PROPOSED ACTIVITY or DISCOVERY create the possibility of a malfunction of ITS EQUIPMENT of a different type than any previously evaluated in the AUTHORIZATION BASIS documentation?

X No ☐ Yes/Maybe

Basis The proposed change is unrelated to any of the Equipment Important To Safety listed in the Authorization Basis. The proposed change could affect only the flow of off-gases through the vertical calciner off-gas system.

- 7 Does the PROPOSED ACTIVITY or DISCOVERY reduce the margin of safety as defined in the basis for any Technical Safety Requirement?

X No ☐ Yes/Maybe

Basis The PFP Operational Safety Requirements do not have associated margins of safety, therefore, those margins can not be reduced by this proposed change.

- 8 Does the PROPOSED ACTIVITY or DISCOVERY require a new or revised Technical Safety Requirement?

X No ☐ Yes/Maybe

Basis The proposed change is not part of any postulated abnormal occurrences or accidents studied in the Authorization Basis, nor are there any Operation Safety Requirements associated with the vacuum controller, therefore, neither new nor revised Operational Safety Requirements are needed.

USQE #1 J A Compton

USQE #2 J P King

(Print Name)

(Print Name)



Signature

Date 1-17-00



Signature

Date 1/17/00

PRC REVIEW (If Required)

Meeting No _____ Date _____

PRC has determined the Proposed Activity or Discovery involves a USQ

Yes No

PRC Chairman _____ Date _____
Signature

DISTRIBUTION SHEET

To
Pu Process Support Labs 1AF00

From
Pu Process Support Labs 1AF00

Page 1 of 1

Project Title/Work Order

Date 1-28-00

Test Plan for Radioactive Testing of a Vertical Direct Denitration
Calciner

EDT No N/A

ECN No 656221

[illegible]

Test Plan for Radioactive Testing of a Vertical Direct Denitration Calciner

J A Compton

Fluor-Daniel Hanford Company

Richland WA 99352

U S Department of Energy Contract DE-AC06-96RL13200

EDT/ECN ECN 656221

UC 2050

Org Code 1AF00

Charge Code 110890

B&R Code EW04J1050

Total Pages 35

Key Words Test Plan Direct Denitration Vertical Calciner, Pu Solutions

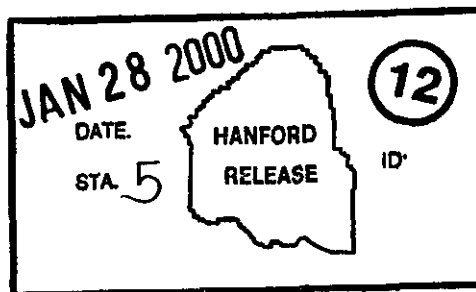
Abstract A vertical calciner will be used to demonstrate the direct denitration process for converting plutonium-bearing liquors to stable plutonium-rich solids. The calciner and some of its ancillary equipment were previously tested with non-radioactive chemicals to demonstrate operability.

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[Signature]
1/28/00

Release Approval



Date

	
This Document is UNCLASSIFIED	
Work Product under this requirement is	
<input checked="" type="checkbox"/> UNCLASSIFIED	<input type="checkbox"/> RD
<input type="checkbox"/> CONFIDENTIAL	<input type="checkbox"/> DI
<input type="checkbox"/> SECRET	
<i>[Signature]</i>	01/28/00
Signature	Date
AUTHORIZED CLASSIFIER	

Release Stamp

Approved For Public Release

RECORD OF REVISION		(1) Document Number HNF-SD-CP TP-088	Page 1
(2) Title Test Plan for Radioactive Testing of a Vertical Direct Denitration Calciner			
CHANGE CONTROL RECORD			
(3) Revision	(4) Description of Change Replace Add and Delete Pages	Authorized for Release	
		(5) Cog Engr	(6) Cog Mgr Date
0	(7) EDT 160207 August 25 1995 (Initial Issue)		
1	Revision 1 of document incorporates ECN 191542 and call out of valve labels	FD Fisher	CS Sutter
2	Revision 2 of document includes design modifications radiological control requirements and incorporates ECN 191543 ECN 191541 and ECN 191522	FD Fisher	CS Sutter
3	Revision 3 of document includes design and Criticality Prevent Specification (CPS) modifications and incorporates ECN 191523	FD Fisher	CS Sutter
4	Revision 4 of document includes additional procedural steps and safety discussion for new equipment added to prevent sudden large amounts of solution from entering the calciner A new section (§7 5) was added to include steps for shutting down the system during emergency conditions This revision incorporates ECN 191544	FD Fisher	CS Sutter
5	Revision 5 of document includes steps for the operation of the purgers This revision incorporates ECN 629407	FD Fisher	CS Sutter
6	Complete rewrite due to revised scope of testing and incorporation of additional safety controls Changes incorporated by ECN 629408	JA Compton	CS Sutter
7	Added step to §7 2 to test operability of a check valve Added steps in §7 3 to check heater controller for proper heating rate and to check operability of the Emergency Shutdown button Renumbered steps in §s 7 2 and 7 3 to incorporate new steps Changes incorporated by ECN 629409	JA Compton	CS Sutter
8	Revised § 4 1 to state that compressed air now comes from a dedicated compressor Revised Table 4 to use a simpler method for requesting the calciner high temperature alarm operability check Changes implemented via ECN 629411	JA Compton	CS Sutter
9	Added §6 7 for Fire Safety Renumbered Operational Safety Compliance section as §6 8 Revised Tables 3 5 and procedure sections to clarify that restrictions in Tables 3 5 do not apply to non fissile feeds Revised procedure sections to implement changes needed from recent testing experience Changes incorporated via ECN 629412	JA Compton	CS Sutter
10	Made editorial changes to alter names of working groups to match new titles per PFP Redesign Changes incorporated per ECN 656220	JA Compton	CS Sutter

[illegible]

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TEST PLAN FOR RADIOACTIVE TESTING OF A VERTICAL DIRECT DENITRATION CALCINER

1 INTRODUCTION

Stored solutions containing plutonium and nitric acid and possibly uranium thorium and minor amounts of other substances will be used for development and demonstration of a vertical calciner direct denitration process for conversion of those to stable storable PuO_2 rich solids. Some of those solutions are quite dilute and very impure these require either pretreatment to make them suitable for calciner feed or an alternate stabilization method. Untreated scrap solutions containing some amounts of sulfate phosphate sodium and/or potassium may also be tested for suitability of direct denitration for conversion directly to PuO_2 -rich solids.

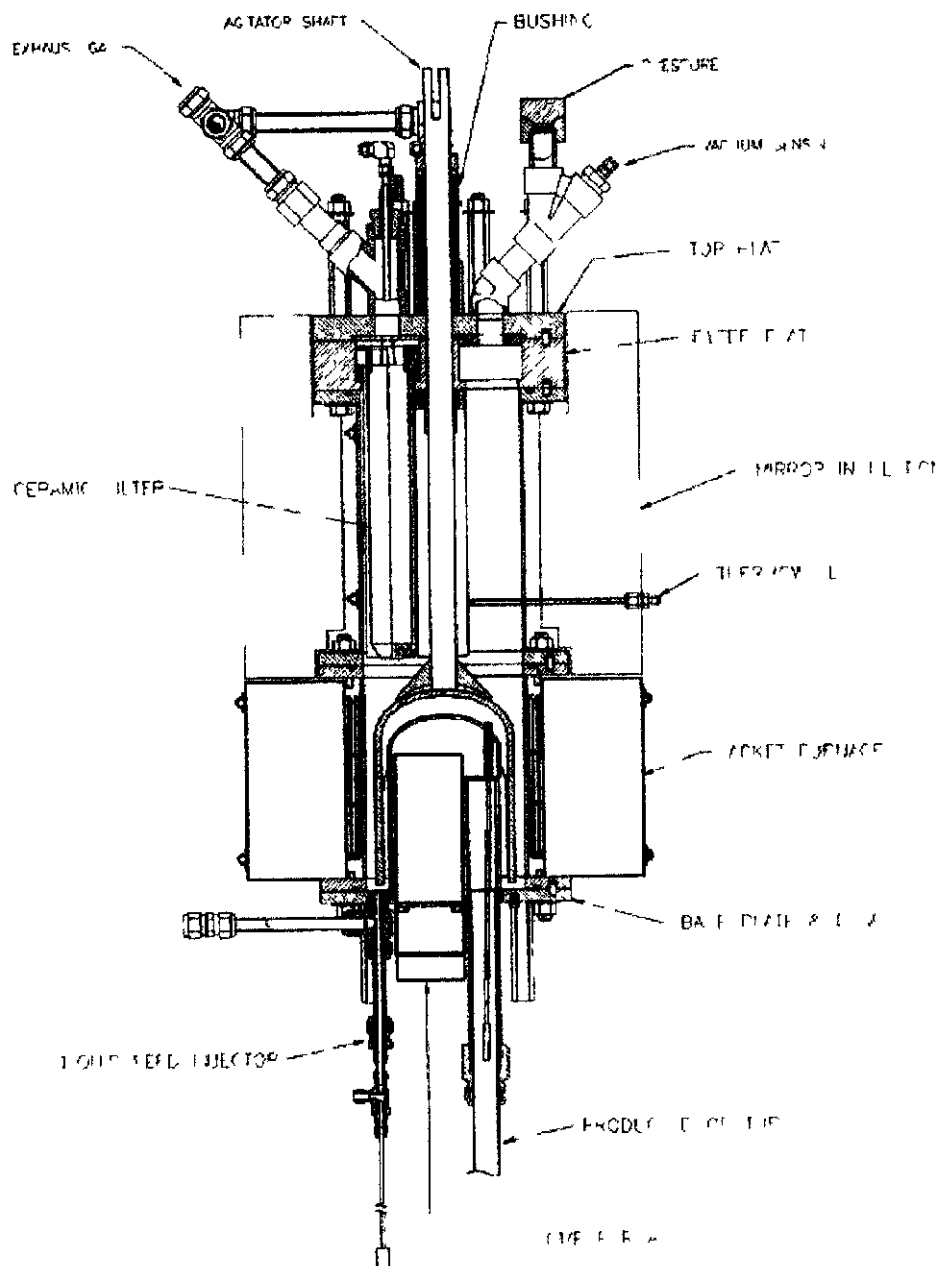
In the vertical calciner direct denitration process to be examined, small additions of liquid feed are metered into a continuously heated and stirred bed of previously generated product solids. The liquid feed is rapidly evaporated then more slowly, undergoes drying and denitration and final heat treatment to stable PuO_2 . The PuO_2 product may contain some residual sulfate and/or phosphate-derived impurities but will be substantially free of chloride, fluoride and other volatile acid impurities if any are present in the feed. Off-gas condensates are expected to be very low in plutonium content. The process is known to work with plutonium thorium uranium and mixtures of those elements in concentrations ranging from 15 to 500 g/L. That range may be extended down to ca 5 g/L. Figures 1 and 2 are a schematic diagram of the vertical calciner and a typical direct denitration flowsheet respectively. Table 1 includes the typical stream values for the flowsheet at the design feed rate of 4 L/hr.

Problems include materials of construction resistant to abrasion and to HCl and HF at 500° 1000°C. Since there is no continuous liquid phase corrosion is limited to vapor phase attack. Ordinary stainless steel may be adequate for the short term although it would exhibit some oxide scaling on the hotter portions of the calciner. A somewhat higher alloy will be more resistant to oxide scaling. The abrasion problem is not extremely severe and, as shaft seals submerged in the powder bed are avoided by design austenitic alloy construction will be adequate. Hard facing of impellers is not believed to be necessary. Suitable bearings for the impeller shaft present formidable materials problems. Graphite is in place now and is expected to wear and in the case of the lower bearing, to experience some oxidation. Frequent inspection and occasional replacement may be required.

The process is not particularly energy efficient for very dilute feeds and cannot handle feeds high in sodium potassium or other constituents that form nitrates that are molten and refractory at $\leq 1000^\circ\text{C}$ such as acid solutions that have been neutralized with NaOH or KOH. Organic impurities are largely consumed and only partly report to the off gas.

A prototype vertical calciner has been built and will be used along with associated ancillary equipment in Glovebox 188 1 to demonstrate direct denitration processing of various plutonium solutions to stable, storable PuO_2 -rich solids. Prior to that this calciner was temporarily installed in Hood 202 1 and operated "cold" i.e., with non-radioactive stand in materials to demonstrate adequate performance of the calciner and most of its associated ancillary equipment items and systems.

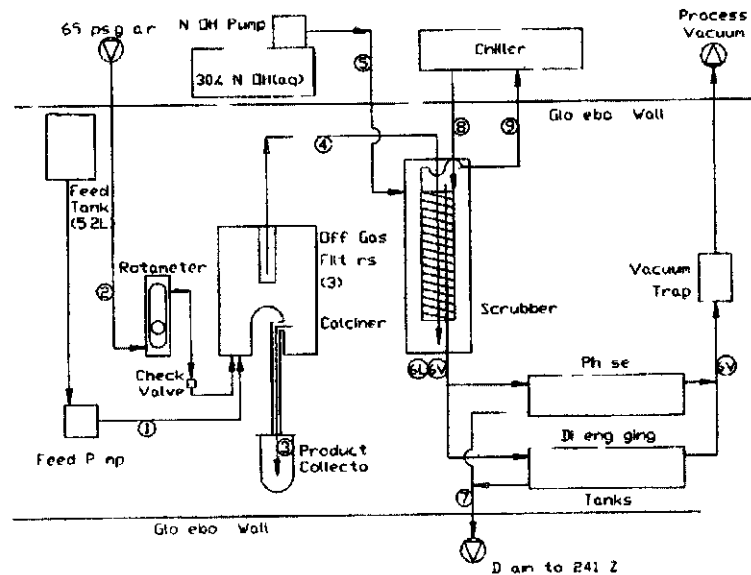
Figure 1 Schematic Diagram of Vertical Calciner



2 OBJECTIVE

- 1 To demonstrate satisfactory direct denitration conversion of plutonium bearing solutions to stable solids in a vertical calciner and ancillaries system
- 2 To demonstrate the adequacy of performance of the crucial components of a vertical calciner and ancillaries system for direct denitration processing of a variety of Pu-bearing solutions and to determine deficiencies for possible remediation

Figure 2 Typical Direct Denitration Flowsheet



3 SCOPE

A production-scale vertical calciner and its ancillary equipment will be operated by the Plutonium Process Support Laboratories for several hundred hours with Pu bearing solutions in Glovebox 188-1. The primary source of calciner feed is product from operation of either the Plutonium-Uranium Extraction (PUREX) Plant or the Plutonium Reclamation Facility (PRF). Further testing will be with other plant solutions.

4 DESCRIPTION OF TEST

4.1 Test Items

The primary test item is the vertical calciner which is built from high chromium high nickel Type 310S stainless steel and field insulated. Figure 1 shows the cross-sectional schematic of the calciner. The calciner has an annular design with a 6 inch sch 10 pipe outer shell and an inner shell of 4 inch pipe. The top of the inner shell is a dome shaped pipe cap. Solids inside the calciner are mixed and moved by a 4-tine agitator that fits inside the annulus covers the dome and extends out the top center of the apparatus. A chain drive connects the top of the agitator shaft to an offset drive motor. A filter chamber extends above the dome and contains 3 blowback filter elements and a pressure relief device. The agitator shaft is supported in the top plates by two graphite or similar bushings and a stuffing box gland.

Crucial supporting equipment used with the calciner includes an off-gas scrubber, feed tank and metering pump, scrub solution tank and metering pump, phase separators, a vacuum trap, and an air-cooled chiller. All these items except the scrub solution tank/pump and the chiller are installed along with the calciner in Glovebox 188-1. Figure 2 is a schematic arrangement of this equipment. Process flow rates and operating conditions are listed for an example flowsheet in Table 1.

TABLE 1
FLOWSHEET PROPERTIES AND VALUES

Property	Units	1 Feed Solution	2 Atomizing Air	3 Product	4 Filtered Calciner Off-Gas	5 Fresh Scrub Solution	6V Scrubbed Off-Gas	6L Spent Scrub Solution	7 Spent Scrub Disposal	8 Cooling Water In	9 Cooling Water Out
Temperature	°C	25	25	650	330	25	21	21	24	7	13
Abs Pressure	atm.	2	5.42	0.83	0.73	1.1	0.66	0.66	1	2.36	2
	Torr	1520	4119	631	555	836	502	502	760	1794	1520
Rel Vacuum	-in H ₂ O	N/A	N/A	69	110	N/A	138	138	0	N/A	N/A
Spec Gravity	gm/cc	1.526	0.0066	5.5	0.0004	1.33	0.0008	1.15	1	1	1
Liq Flow Rate	L/hr	4	N/A	N/A	N/A	4.42	N/A	8.73	Batch	659	659
Pu Concent'n	gm/L	234	N/A	4750	N/A	N/A	N/A	N/A	N/A	0	0
Pu Mass Rate	gm/hr	936	N/A	936	0	0	0	0	0	0	0
Pu Mole Rate	mole/hr	3.92	N/A	3.92	0	0	0	0	0	0	0
PuO ₂ Rate	gm/hr	N/A	N/A	1061	0	0	0	0	0	0	0
HNO ₃ Concent'n	mole/L	5.68	N/A	N/A	N/A	N/A	0	0	0	0	0
HNO ₃ Rate	mole/hr	22.72	N/A	N/A	22.72	N/A	0	0	0	0	0
Water Concent'n	mole/L	38.4	N/A	N/A	N/A	51.6	N/A	46.7	46.7	55.5	55.5
Water Rate	mole/hr	153.5	N/A	N/A	N/A	228.1	N/A	408	Batch	36,575	36,575

TABLE 1
FLOWSHEET PROPERTIES AND VALUES

Property	Units	1 Feed Solution	2 Atomizing Air	3 Product	4 Filtered Calmer Off-Gas	5 Fresh Scrub Solution	6V Scrubbed Off-Gas	6L Spent Scrub Solution	7 Spent Scrub Disposal	8 Cooling Water In	9 Cooling Water Out
Gas Flow Rate	scfh	N/A	40	N/A	189.2	N/A	42.7	N/A	N/A	0	0
	std L/hr	N/A	1132	N/A	5353.7	N/A	1209.6	N/A	N/A	0	0
Oxygen Rate	mole/hr	N/A	10.6	N/A	13.1	N/A	13.1	N/A	N/A	N/A	N/A
Nitrogen Rate	mole/hr	N/A	39.9	N/A	39.9	N/A	39.9	N/A	N/A	N/A	N/A
H ₂ O Vapor Rate	mole/hr	N/A	N/A	N/A	153.5	N/A	0.8	0.8	0	0	0
NO Gas Rate	mole/hr	N/A	N/A	N/A	0	N/A	0	N/A	N/A	N/A	N/A
NO ₂ Gas Rate	mole/hr	N/A	N/A	N/A	9.8	N/A	0.2	N/A	N/A	N/A	N/A
NaOH Concentr'n	mole/L	N/A	N/A	N/A	N/A	10	N/A	1.32	1.32	N/A	N/A
NaOH Rate	mole/hr	N/A	N/A	N/A	N/A	44.2	N/A	11.5	Batch	N/A	N/A
NaNO ₃ Concentr'n	mole/L	N/A	N/A	N/A	N/A	N/A	N/A	3.17	3.17	N/A	N/A
NaNO ₃ Rate	mole/hr	N/A	N/A	N/A	N/A	N/A	N/A	27.7	Batch	N/A	N/A
NaNO ₂ Concentr'n	mole/L	N/A	N/A	N/A	N/A	N/A	N/A	0.57	0.57	N/A	N/A
NaNO ₂ Rate	mole/hr	N/A	N/A	N/A	N/A	N/A	N/A	5.0	Batch	N/A	N/A

The calciner control panel and seismic shutdown equipment are located outside the glovebox. The seismic shutdown equipment shuts down all electrical power to the control panel and glovebox plus the pressurized air supply into the glovebox. This shutdown equipment acts on a signal from two accelerometers elsewhere in the building.

Building process vacuum, pressurized air, and electricity are also used by the calciner system. The vacuum is the nominal 26-inch mercury vacuum supply. Pressurized air is supplied to the room from a dedicated compressor at a nominal 90 psig and reduced as needed within the room. Electricity is supplied to Panel AZ within the room from the 480 VAC Panel PB in the duct level.

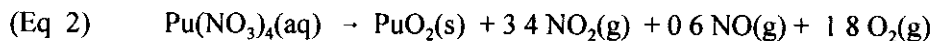
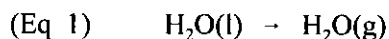
Drawing numbers for the crucial equipment pieces and installation are given in Table 2.

TABLE 2
PRIMARY EQUIPMENT DRAWINGS

<i>Equipment/Installation Description</i>	<i>Drawing Number(s)</i>
<i>Calciner</i>	
Calciner/Filter/Agitator	SK 2-300303
Agitator Drive Assembly	SK 2-300305
Product Collector	SK-2-300305
Filter Blow Back Apparatus	SK-2 300305
<i>Crucial In Glovebox Equipment</i>	
Scrubber	SK 2-300306
Feed Tank	SK 2-300307
Effluent Collection Tanks	SK-2-300308
Vacuum Trap	SK-2 300310
Piping & Instrumentation Diagram	H-2-829518
<i>Glovebox Equipment Arrangement</i>	SK 2 300304
<i>Electrical/Instruments Installation</i>	SK 2-824342
<i>Seismic Shutdown Installation</i>	H-2 28551

Other items included with the equipment listed above are the three 6 point temperature recorders, both calciner heaters with their power supplies and controllers, a starting bed of plutonium dioxide powder, and a feed stream of plutonium nitrate in nitric acid solution. Additional chemicals may also be present in the feed and powder bed.

Plutonium nitrate solution undergoes evaporation and thermal decomposition in the heated stirred bed vertical calciner largely according to



The solid plutonium dioxide formed increases the bed volume, causing solids to overflow to a product receiver vessel below the calciner.

The scrubber absorbs gases into a caustic solution that is maintained at about 1.5 M NaOH. Fresh scrub solution of 10 M NaOH (30 wt %) is metered into the scrubber continuously to maintain the desired steady state NaOH concentration. Spent scrub solution overflows continuously with the scrubbed gases to the phase disengaging tanks. The scrubber normally operates at about 20°C due to the cooling water flow. The cooling water inlet temperature is typically 7 °C and the outlet temperature is typically 13°C.

4.2 Test Environment

The testing will be performed in Glovebox 188.1 in Room 188 of the 234-5Z Building, 200 West Area. Control and measurement equipment will be arranged around that glovebox as appropriate. The glovebox provides confinement of the chemicals in use. The glovebox will breathe room air through five 8"x8"x4" inlet filters and will exhaust through three like filters to the E-4 system.

4.3 Equipment and Facilities

Equipment other than that being tested includes normal glovebox supporting equipment: an electronic balance, canning supplies and machines, and 4-liter or smaller bottles of plutonium nitrate solution for feed.

Other facilities to be used include Room 190 for vault storage of product and the building radioactive drain system for spent scrub solutions.

4.4 Data

Temperatures to be measured and recorded include:

- ◇ Calciner exterior wall (2 locations: 1 for control and 1 for high temperature alarm)
- ◇ Calciner dome
- ◇ Calciner base plate (3 locations: including next to feed port)
- ◇ Inside the calciner/filter chamber
- ◇ Filter chamber exterior (2 locations)
- ◇ Tubing exterior of filtered exhaust (2 locations)
- ◇ Glovebox interior (at E-4 filters)
- ◇ Scrubbed exhaust gases
- ◇ Scrubber cooling water inlet and outlet
- ◇ Drive motor exterior

Due to aging, wear, and difficulty of replacement, not all thermocouples will remain working.

Pressures to be measured and recorded periodically are the pressure difference across the blowback filter elements and the vacuum level inside the calciner filter chamber. All pressure recording will be manual. Additional vacuum gauges are present throughout the off-gas system to aid in troubleshooting if off-gas flow problems develop.

The only flow rates measured directly are the pressurized air flow into the calciner at the feed port and a purge air flow rate used to keep the calciner vacuum indicator (PI-2) sensing line clean, both via rotameters. Times and solution levels inside the feed tank will be used to check the metered feed flow rate. Scrub solution metered flow rates are estimated by observing the liquid accumulation rate in the phase disengaging tanks.

Feed volumes and plutonium concentrations will be known before entry into the glovebox. Product weights will be obtained via the electronic balance inside the glovebox. Product plutonium contents will be determined by calorimetric measurements in 2736 ZB. They will be estimated for transfer purposes prior to calorimetry as the best estimate of a fraction of the product net weight (these fractions will be determined by the previous calorimetry).

and net weight results then adjusted for the relative purity of the new feed solution) Records of these amounts will be kept in the laboratory notebook

4.5 Criteria/Constraints

An addendum (Reference 1) to the PFP Final Safety Analysis Report (FSAR) (Reference 2) was prepared specifically for this calciner. This addendum includes a thorough hazard analysis to ensure that there are no Unreviewed Safety Questions (USQs) possible for the testing of this calciner within the limits given in Section 6.8 of this document. Unreviewed Safety Question Screening 99-014 confirms that there are no USQs.

4.6 Test Design

The calciner will be tested in a series of runs of varying length. The first set of runs will be short runs using dilute acid solution as feed. These tests will be used to check equipment operability and to educate staff on most aspects of calciner operation.

Those runs will be followed by a series of short runs using plutonium nitrate solution as feed. These runs will be used to finish educating the staff on calciner operation including removal of product and staying within criticality dispersibility and security limits with plutonium. The average residence time will be varied within the runs to determine its effect on product quality. Different average residence times can be achieved by altering the volume of the powder bed within the calciner, the solution feed rate, and/or the concentration of plutonium in the feed. The plutonium concentration is certain to change at least slightly from run to run.

Tests with varied parameters are normally run in a random pattern; however, randomness will be difficult to achieve most of the time. The powder bed volume in the calciner will not be adjusted randomly due to (1) the maintenance and related radiological exposure for changing the amount of powder in the bed and (2) the uncertainty of repeating the different powder levels accurately. The need to make consecutive runs from the same feed batch limits the ability to vary the feed concentration randomly.

The total number of short runs needed can not be estimated due to uncertainties with staff scheduling, the results from each run, and the (subjective) degree of learning achieved during each short run. One goal is to have each person who will work on longer runs assist with at least two short runs in this phase.

The third set of runs will be a mixture of longer runs and short runs, if possible. The longer runs are intended to feed solution to the calciner for at least 20 hours to determine if longer runs affect the calciner components adversely. The number of longer runs can not be estimated due to uncertainties with staff availability, test results, and potential interferences with running other vital projects.

5 EXPECTED RESULTS

The calciner and its associated ancillaries are expected to function smoothly and adequately and that the design throughput capability of 4 L/hr of feed solution will be achieved. The calciner is expected to operate smoothly and efficiently when solution is fed to it continuously for at least 20 hours. The calciner and ancillaries are expected to withstand corrosive attack by the acid mixtures in the feeds through the life of the tests. Products from this calciner are expected to exhibit a Loss on Ignition (LOI) of <0.5 weight percent at 950°C. These results are expected with moderately impure solutions and with relatively pure solutions.

6 SAFETY

Automated Job Hazard Analysis (AJHA) number 2Z 294 has been completed and approved for this test plan. This AJHA is an integrated computer maintained form that addresses all potential types of hazards and assigns any necessary controls for identified hazards. Required approvals are also assigned automatically.

A Hazards and Operability study specifically for this calciner has also been performed by Safety Analysis personnel in Fluor Daniel Northwest, Inc. This HazOp analysis is included in the FSAR addendum (Reference 1).

6.1 Criticality Safety

Criticality Safety Evaluation Report (CSER) 99-001 (Reference 3) and Criticality Prevention Specification (CPS) CPS-L 114 00050 were written exclusively for these tests. This CPS allows the glovebox to contain a plutonium amount adequate for all planned testing needs while maintaining the plutonium in a non-critical state under all credible processing and upset conditions, including an earthquake. Important restrictions in addition to the mass limit include:

- ◇ A maximum product tap density of 5.5 gm Pu per cc of powder. This density must be checked at the end of each run. The densities will be recorded and trended to allow ceasing operations before the density limit is exceeded. Corrective actions to avoid exceeding this limit will be approved by the PFP Criticality Safety Representative (CSR).
- ◇ Only one feed container may be inside the glovebox at any time, empty or full. A 0.5 L polyjar of water for flushing the feed line is considered a feed container.
- ◇ Only four 0.5-L slip-lid cans or polyjars may be in the glovebox at the same time. Furthermore, no more than two of these may be in the glovebox 'attic' section; only one may be on the glovebox main floor; and no more than two may be in the glovebox 'basement' section. Any containers in the attic are forbidden to contain liquids.
- ◇ Fissile material containers must be kept at least 10 inches from each other and from fixed equipment containing fissile material except when transferring between containers. Allowed transfers between containers are from feed bottle to feed tank, from product collector to product can, between 0.5 L containers from product can to the calciner filter chamber (to return powder to the powder bed), and from a container into a sample bottle. Containers may not be placed onto any horizontal surface of the calciner assembly, including the skirting around the exterior heaters.
- ◇ Only 20 sample bottles are allowed inside the glovebox at any time; no more than 10 of those may be grouped together.

Strict adherence to this CPS and related postings will be maintained. Monthly internal inspections by PPSL personnel and additional inspections by the PFP CSR serve as additional compliance checks.

The equipment arrangement design was studied and approved by the CSR prior to issuance. Any changes to this design must be approved by the CSR.

The cooling water system for the scrubber presents another theoretical criticality prevention problem not completely observable from within Room 188. The cooling water loop contains about 20 L of water, much of which is in a reservoir inside the chiller in Room 187. The reservoir geometry is not favorable for criticality prevention. A criticality is extremely unlikely in the reservoir for five reasons. First, there would have to be simultaneous leaks in the cooling water coils inside the scrubber and at least one blowback filter element to allow plutonium into the cooling water. Second, the cooling water is pressurized while the scrubber is under vacuum; therefore, the leak path would be from the cooling water coils into the contaminated scrub solution until the cooling water loop is nearly empty. Third, the workers would then have to fail to notice that the cooling system was not operating correctly to cool the scrub solution and that cooling water was being forced from the cooling coils into the scrub solution. Fourth, the workers would have to fail to notice the darkening green color of the scrub solution as the

plutonium concentration increased to levels that would cause concern for criticality in the reservoir. Finally, the scrubber operates with a constant overflow to the "geometrically favorable" phase disengaging tanks. This overflow solution would carry some of the plutonium with it, reducing the amount of plutonium that could eventually get into the chiller reservoir. Room 188 must be occupied by at least two workers any time the calciner is running.

6.2 Radiological Safety

All work associated with this project will be performed in accordance with the applicable Radiation Work Permits (RWPs). In addition, the dose rates on the glovebox are measured and posted on the glovebox routinely by Radiological Control Technicians (RCTs). The RCTs also routinely check air samplers and Continuous Air Monitors (CAMs) for proper operation and satisfactory work conditions. An RCT must also be present whenever items are moved into or out of the glovebox, except for moving items into the glovebox via the sphincter port.

The recirculating cooling water between the chiller and scrubber could leak from the tubing. The volume of water in the closed cooling loop is finite and fairly small (about 20 L). The cooling water is theoretically sealed from contamination sources; however, leaks of contaminated scrub solution into the cooling water are possible. The scrub solution should not be extensively contaminated, though, unless at least one blow back filter element also is breached. Consequently, any leaks should be not only small in volume but also low in contamination. Any leaks would also be very unlikely to cause an airborne contamination problem because these leaks would be from a liquid solution. Additionally, the chiller, scrubber, and connecting tubing are all completely within posted Contamination Areas.

Work involving the use of pressurized air inside a glovebox presents the theoretical possibility of pressurizing the glovebox. Glovebox 188-1 was analyzed for potential pressurization with air supplied to the glovebox at up to 90 psig. Pressurization was determined not to be possible beyond levels normally encountered across the glovebox wall, but in the opposite direction. If both pressure regulators in the air supply line failed simultaneously and made 90 psig air available to the glovebox, the maximum flow rate would be only about 82 standard cubic feet per minute (scfm). If the glovebox ventilation is also lost at the same time, the corresponding pressure inside the glovebox would still be only about 1.5 inches of water, which is well within the normal operating range for the pressure difference across the inlet HEPA filters, gloves, and windows (Reference 3). Since this analysis was performed, the inlet air filters have been increased from 3 to 5, further reducing the potential for breaching the glovebox.

Plutonium nitrate solution or other forms of plutonium are prevented from entering the pressurized air line to the glovebox by check valves. The feed nozzle area of the calciner contains a check valve just before the air and feed solution enter the calciner. A small rotameter is placed just upstream of the check valve in this branch of the air line to allow a visual indication if any solution is being forced back up the air line. The incoming air pressure is also typically 60-65 psig, which is higher than the pressure of the feed solution line. If the air pressure drops below 60 psig, the feed pump shuts off automatically. A solenoid valve is also located in this line. This solenoid valve requires power to open and power is shut off to this valve when the calciner is not being used. Plutonium dioxide powder can not enter the pressurized air line outside the glovebox because of two check valves in that portion of the air line. In addition, two solenoid valves would have to fail. These solenoid valves also require power to open and are shut off automatically when the calciner is shut down.

A check valve has been placed in the supply line for fresh caustic solution being fed to the off-gas scrubber. This check valve is just inside the glovebox and prevents contaminated scrub solution from being siphoned out of the scrubber and into the room through a leak in the caustic supply line when the caustic supply pump is shut off. The fresh caustic supply itself is further protected by a manual valve and two check valves in the pump when the pump is shut off.

Post-job As Low As Reasonably Achievable (ALARA) Reviews are required after the first calciner run and again within the first 3 months of testing. Results from these reviews will be used to determine if changes are needed in any procedures and/or in the RWPs. The first review will include the collective dose data from the first run.

The prototype calciner is connected to the building seismic event detector. Power shuts off automatically via the Supply Ventilation System Seismic Shutdown if an acceleration of 0.07 g is detected. Loss of main power shuts off the feed pump, agitator, filter blowback solenoid valves, and the pressurized air supply solenoid valves. With those components shut off, there is no way to pressurize or agitate the calciner and force PuO_2 out of the calciner and into the glovebox or room. Keeping the PuO_2 inside the calciner body minimizes any spread of dispersible plutonium to the room, building occupants, or the public if an earthquake occurs.

6.3 Industrial Safety

This testing involves high-temperature, electrically heated chemical processing equipment. The thermally hot equipment is extensively insulated and hot surfaces are difficult to reach. The highest exterior temperature recorded during non-radioactive operation was 92°C on the agitator shaft where it extends out from the calciner, which is an extremely difficult location to reach inside the glovebox. Temperature resistant Kevlar¹ Hot Mill gloves rated for use up to 450°F are available and will be worn if reaching near the hot surfaces is required. Maintenance work will be performed after the calciner has had ample time to cool, if at all possible. Power to the heaters shuts off immediately via the building seismic event detector if an earthquake occurs. Loss of heater power aids in cooling the calciner more quickly to reduce the chance of igniting anything coming in contact with newly exposed hot surfaces.

The calciner and scrubber are tall, heavy pieces of equipment and require supports to maintain their upright orientation. The needed supports are provided and are adequate to prevent the equipment from tipping. A hoist is provided in the glovebox to assist with maintenance activities when needed.

The only exposed rotating part is the top of the agitator shaft, which is difficult and unnecessary to reach during normal operation. The exposed portion contains no sharp or protruding edges. Other pieces of moving or rotating equipment, most notably the agitator drive system, are shielded from workers by guards and/or are difficult to reach during normal operation. The provisions of *Machine Guarding* (HNF-PRO 086) will be used to maintain safety during maintenance involving rotating or moving parts. The agitator also shuts off automatically due to loss of main power in the room in case of an earthquake.

Viewing or using some of the equipment in the calciner glovebox requires the use of ladders. Two rolling ladders are located inside the room for these purposes. These ladders have metal frames and steps, plus a handrail up both sides and around three sides of the top step. The frame is mounted on weak springs that lift it just above the bottoms of the wheels to allow movement when unoccupied. Any mild force pushing downward on the springs places rubber-covered feet onto the floor to prevent further movement.

Tasks involving respirators present a noise hazard in addition to radiological hazards. The noise generated by the air sampling machines during and after the work tasks can make verbal communication difficult. Methods to minimize the communications difficulties will be addressed at each pre-job meeting.

¹ Kevlar is a registered trademark of E. I. duPont de Nemours and Co., Inc., Wilmington, DE.

6.4 Electrical Safety

All equipment is installed according to designs approved by the appropriate safety organizations and per *Electrical Installation Safety* (HNF-PRO-089). Electrical connections are tested for proper installation prior to starting these tests. Bare electrical connections will not be accessible to casual contact. The provisions of *Electrical Work Safety* (HNF-PRO 088) will be used to maintain safety during maintenance involving electrical parts. The main power through Panel AZ is lost automatically in case of an earthquake.

6.5 Chemical Safety

Most chemical operations occur inside the glovebox, which shields workers from chemical hazards. Protective clothing required by the RWPs and the glovebox ventilation system serve as additional barriers to chemical exposure. Chemical safety outside the glovebox is assured by rigid adherence to the PPSL Chemical Hygiene Plan (Reference 4). The MSDSs for all non-radioactive chemicals are available electronically through the Hanford Intranet.

Due to the high operating temperature reached prior to feed commencement, any sudden "large" amounts of feed entering the calciner will vaporize rapidly, possibly overwhelming the off-gas system's ability to remove the vapors as they form. The introduction of a large amount of feed suddenly is most likely to occur when clearing a solid plug in the feed nozzle if the plug is not noticed quickly. A plug would most likely be noticed in one of two locations. The plug might be noticed by a lack of variance in the indications on the calciner pressure gauge. The gases formed with each feed pulse yield definite transient changes in calciner pressure and gauge indication. The plug might also be noticed at the rotameter for the incoming pressurized air line. A plug would try to direct any incoming feed solution back up the air line and this would stop air flow through the rotameter. A check valve in the air line prevents the feed solution from reaching or going past the rotameter and contaminating the inside of the non-radioactive pressurized supply line outside the glovebox.

If a plug occurs, the pressurized air supply drives any accumulating solution in the annulus around the feed line up into the calciner immediately when the plug is cleared. Due to the possibility of forming so much vapor so rapidly in the calciner, the Procedure section contains steps to minimize the possibility of pressurizing the calciner. Plugs may only be removed by workers during operation if the plug is noticed quickly enough to keep the accumulated solution volume small. If the accumulated solution volume is thought to be too large for the calciner to handle all at once, the system is shut down so that Maintenance can remove the feed injection nozzle to clear the plug.

Overpressurization of the calciner from chemical reactions or excessive feed flow is minimized by a pressure relief device. The device is a 3/4-inch hole at the top of the calciner with a cap held on by gravity and beveled edges. The cap will be lifted upward to relieve pressure starting at 1.3 psig. Experience during testing has confirmed that the pressure relief device works as intended.

The pressure relief device can vent all the gases generated in or introduced into the calciner at any realistic rate at well below the pressure required to rupture the calciner, i.e., about 300 psig, even with the off-gas system sealed off. The feed pump will not inject feed at pressures above 55 psig. The atomizing air and blowback air are normally 60-65 psig, and the ultimate pressure available to both of those is only 90 psig. A steam surge from siphoning back of scrub liquor into a hot calciner would be limited by pressurization terminating the siphoning after only a small amount of scrub liquor was introduced to the hot calciner. This pressure would reverse the scrub liquor flow back to the scrubber and the pressure would then relieve through the device hole.

Three methods are used to prevent organics from entering the calciner, where they might combust and overpressurize the calciner. First, the download procedure for removing the solutions from their original containers requires a visual check for a second liquid phase. Second, sample analyses by the PFP Analytical Laboratory confirm the lack of organics in the feed samples. Finally, the feed tank in the calciner glovebox is glass, allowing

staff to notice any organic phase in the feed. The calciner can safely accept sub-saturation concentrations of organics and did so in the first run with plutonium feed. Those are promptly combusted upon introduction and, thus, cannot accumulate into hazardous quantities due to their very slight solubilities.

The caustic supply line from the caustic feed pump to the glovebox wall penetration is shrouded in plastic sheeting. The shroud was made by draping a flat plastic sheet over the top of the line and taping the draped ends at spaced intervals. This design turns any sprays of caustic solution into drips that fall harmlessly to the room floor between the pieces of tape. The likelihood of a caustic spray through a leaking fitting or line due to a plugged transfer line is minimized by a pressure relief device built into the outlet of the diaphragm pump for the caustic solution. This relief device reroutes any solutions above a preset outlet pressure back into the caustic supply drum. The preset pressure is 25 psig. If caustic comes in contact with human skin or clothing, anyway, a safety shower is located along the west wall of Room 188.

6.6 Seismic Safety

Calciner conditions during and after an earthquake must remain within approved risk guidelines. The Fluor Daniel Northwest Safety Analysis team postulated that the calciner would exceed approved risk guidelines unless certain safety measures were installed (Reference 1). These safety measures are needed to counteract the expected effects of an earthquake on the calciner and throughout the building. In their worst case scenario, the building loses the process vacuum supply while retaining the pressurized air supply; some or all of the glovebox windows break or pop loose, and the building suffers structural damage at the walls. In addition, combustible materials in the glovebox might contact hot surfaces and start a fire. If vacuum is lost and pressurized air remains available, the calciner will pressurize and blow PuO_2 powder out through the pressure relief port. Some of this powder could be blown or carried out of the glovebox into the room and, eventually, into the environment through cracked walls. The postulated releases inside and outside the building exceed the dose rates allowable from a seismic event to both workers and the public. Three measures were taken to reduce the risk to acceptable levels.

The glovebox was bolted to the room floor in a seismically qualified manner. This step keeps the glovebox from toppling over during the earthquake, which would increase the amount of damage to the glovebox walls and windows. Worse, a toppling glovebox also presents serious physical hazards to workers in the room. This step secured the glovebox itself, but did not secure the equipment inside the glovebox.

Seismically qualified solenoid valves were added to the pressurized air supply line outside the glovebox. These valves shut off automatically during an earthquake to reduce the potential for pressurizing the calciner through the feed injection port. Once inside the glovebox, the air supply line splits into two branches directed toward the feed injection port area and the blowback filter system. Two check valves in series were added to the branch going to the blowback system's accumulator tank. These check valves ensure that the pressurized air already in that tank can not flow back into the other branch and reach the feed injection port area to pressurize the calcine briefly that way. A solenoid valve that requires power to open is also present at the beginning of the branch going to the feed injection port area. This valve should also close on loss of power as another means of preventing the accumulator tank from emptying into the bottom of the calciner. These check valves and solenoid valve are not seismically qualified, however, tests have shown that the accumulator tank loses its air supply completely in only 15-16 seconds if all three of those valves fail to work properly. The air pressure and flow rate decrease rapidly and steadily during those 15-16 seconds.

Finally, power to the calciner control panel was connected to the building seismic shutdown system to cut off all power to the calciner system if an earthquake causes a floor acceleration of as little as 0.07 g. The power disconnect is made by using seismically qualified relays and contacts in series to guarantee that the power will be lost reliably when an earthquake strikes (Reference 5). The signal that initiates the power loss comes from the accelerometers that also shut down the building ventilation supply fans during a 0.07 g floor acceleration. The loss of power shuts off the feed pump, the agitator, the filter blowback system, the calciner heaters and the caustic

supply pump. Loss of the feed pump prevents the calciner from pressurizing due to the gases that would be formed in the hot powder and also prevents more PuO_2 from being formed. Loss of the agitator prevents the powder bed from being stirred mechanically to loft powder directly. This loss also prevents finer powders that are easier to loft by other means from being made. Loss of the blowback filter system prevents pressurized air from pressurizing the calciner by way of the blowback system. The blowback system uses two solenoid valves in series with one common valve and then one on each of the three branches to the filter elements. All those valves require power to open and the latter valves are only opened during normal use for very brief, well spaced periods. If both valves are open during an earthquake, only the powder on the filter elements being blown back could be lofted into and beyond the glovebox atmosphere. Loss of the heaters allows the system to begin cooling more quickly with a potential reduction in the likelihood of starting a fire. This loss of power also shuts off the lights for the calciner glovebox but has no effect on the room lights.

The Criticality Safety Evaluation Report considered the worst case for the amounts of plutonium, reflectors, and moderators theoretically present and concluded that an earthquake will not cause a criticality in the prototype calciner glovebox.

6.7 Fire Safety

Combustible materials are minimized inside the glovebox per normal practice. Waste solids, particularly combustible solid wastes, are kept near the vacuum trap in the main level of the calciner glovebox. This location keeps the solids well away from the calciner, which is the only heat source, and all electrical apparatus. In addition, the calciner insulation and skirting make contact with hot surfaces extremely difficult. The hottest exposed surface on the calciner is along the joint between the calciner insulation skirting and the filter chamber insulation ("mirror insulation") where the maximum recorded temperature is only 77°C . This temperature is above the normal use limit for glovebox gloves (71°C) but still is not high enough to combust a glove.

A high temperature detector is located in the ceiling of the glovebox near the exhaust HEPA filters and connected to the building fire alarm system. A bayoneted fire extinguisher is in the corridor just outside Room 188, if needed.

6.8 Operational Controls Compliance

Operational controls were included in the addendum (Reference 1) to the Final Safety Analysis Report. The controls directly related to Operational Safety Requirements (OSRs) from Section 5.2.1, "Operational Safety Requirements" of Reference 1, are listed with their implementations in Table 3. Operational controls not associated directly with the OSRs from Section 5.2.2.1, "Operational Controls" of Reference 1, are listed with their implementations in Table 4. Additional controls were imposed in Section 5.2.2.2, "Controls Required to Implement Key Analytical Assumptions" of Reference 1. These controls and compliance with them are described in Table 5.

TABLE 3
OPERATIONAL SAFETY REQUIREMENT COMPLIANCE (Addendum Section 5.2.1)

OSR Requirement	Implementation
1 The automatic seismic shutdown system shall be verified operable before initiating calciner tests	A This test plan, Section 7.1, step 2 B WHC-SD-CP-CP-OSR-010, Rev O-J, <i>Plutonium Finishing Plant Operational Safety Requirements</i> Limiting Condition for Operation 3.2.3 "Supply Ventilation System Seismic Shutdown" and Surveillance Requirement 3.2.3.3, "Verify both seismic shutdown system accelerometers are on line and in service" C ZO 100.403 "Seismic Shutdown Accelerometer Surveillance"
2 The isotopic distribution of the plutonium in the calciner feed shall contain less than 10 percent ²⁴⁰ Pu	A This test plan Section 7.1 step 1, third bullet B WHC-SD-CP-CP OSR 010 Rev O-J <i>Plutonium Finishing Plant Operational Safety Requirements</i> Table 5.22.2 Maximum DISPERSIBLE Plutonium Limits
3 The maximum plutonium inventory in the glovebox shall not exceed 7000 grams	A WHC SD CP CP OSR 010, Rev O J, <i>Plutonium Finishing Plant Operational Safety Requirements</i> Administrative Control 5.22 "Plutonium Inventory" B Criticality Prevention Specification CPS L-114-00050

TABLE 4
OPERATIONAL CONTROLS COMPLIANCE (Addendum Section 5 2 2 1)

Control Subject/Requirement	Implementing Document
1 Fissile feed solutions transported in polybottles in vented Product Receiver (PR) Containers or Isolated Transportation Containers (ITCs)	Work Plan WP-PFP 96 LSS-004, "Load In Solution From PR Container Into HC 227S BT-3"
2 Workers use respiratory protection while unloading PR containers and ITCs loading solutions in and bagging out	A WHC-SD CP-OSR-010 (Reference 5) AC 5 16 <i>Radiation Protection</i> B Radiation Work Procedure (RWP) Z-005
3 Glovebox negative pressure verified before beginning glovebox work	This test plan Section 7 1 Step 5
4 Glovebox ventilation flow verified before starting any glovebox work	This test plan Section 7 1 Step 5
5 Calciner High Temperature Alarm operable	Performed on specific request under standing work order to troubleshoot and repair calciner components (for example, work package 2Z-99-317 for Fiscal Year 1999)
6 Room 188 CAM verified as operable before work in room is started	A WHC SD CP OSR 010 AC 5 16 <i>Radiation Protection</i> B RWP Z-(all)
7 Fire protection measures institutionalized to minimize fire hazard Includes limiting glovebox combustible loading, operability of glovebox high temperature detector in fire alarm system and availability of bayonet fire extinguisher	WHC-SD-CP-OSR 010, AC 5 20, <i>Fire Protection</i>
8 Compressed air oil trap operational and continuously drained	This test plan, Section 7 1 Step 7
9 Controls prevent introduction of incompatible chemicals to feed tank or into calciner that could cause a fire or deflagration	A PFP Analytical Services procedure ZA-519 351, Phase Determination of Samples ' B This test plan Section 7 1 Step 1
10 Controls implemented to ensure safe use and transfer of caustic solution	PFP Administrative Procedure ZAP-000-010 <i>PFP Chemical Hygiene Plan</i>

TABLE 5
KEY ANALYTICAL ASSUMPTION COMPLIANCE (Addendum section 5 2 2 2)

Analytical Assumption	Compliance
1 Transfer and receipt of fissile feed solutions shall be limited to polybottles containing a maximum of 2.5 L of solution in a 3 L bottle to limit the hydrogen generation rate during transfers	Work Plan WP PFP 96 LSS-004 "Load In Solution From PR Container Into HC 227S BT 3"
2 The plutonium nitrate feed solutions shall contain a maximum of 450 gm Pu per liter for criticality control purposes, to limit hydrogen generation and to remain within the bounds of the dose consequence analysis	This test plan Section 7.1 step 1, first bullet There are no known solutions in the PFP inventory with Pu concentrations greater than 450 gm/L
3 Fissile feed solutions shall have a nitric acid concentration greater than 4 M to limit the hydrogen generation rate during container transfers and interim storage, if necessary	This test plan Section 7.1, step 1 third bullet
4 Fissile feed solutions shall be limited to plutonium nitrate Other materials have not been evaluated for processing in the calciner	This test plan, Section 7.1 step 1, fourth bullet
5 The calciner feed solution shall be limited to 1% organic to limit the potential for an organic burn in the calciner Feed solutions shall be tested for a visible layer of organic Solutions with separable organic shall not be processed through the calciner	This test plan, Section 7.1, step 1 fifth bullet This test plan Section 7.3 step 9
6 The maximum glovebox inventory shall not exceed 7000 gm Pu to meet the analytical assumption for material at risk in a seismic event	A WHC-SD-CP-CP-OSR-010 Rev O-J, <i>Plutonium Finishing Plant Operational Safety Requirements</i> Administrative Control 5.22 "Plutonium Inventory" B Criticality Prevention Specification CPS-L-114 00050

7 TEST PROCEDURE

The instructions below are written from past test experience with this calciner and engineering judgement. Unexpected problems or situations can occur. When these problems/situations are noticed, consult the responsible engineer for the appropriate response. In all cases, shutting down the calciner system via the button-shaped Emergency Shutdown Switch is always an acceptable response. Whenever necessary to shut down the vacuum system, always shut off the pressurized air first.

7.1 Prerequisite Checks

The following items must be checked on the day before the planned run for any test run using plutonium or any operability check where fissile material will be moved. The items below may be checked in any order. Postpone the run if any of the following items can not be met or is thought improbable to meet. The feed criteria in Step 1 do not apply when using just acid solutions as feed.

- 1 Confirm with Solution Handling (or other group with feed for the calciner) that feed solution is available suitable and that an authorized Nuclear Material Custodian will be available for the transfer papers
 "Suitable feed is feed solution that meets
 - Pu concentration less than 450 gm/L (there are no known solutions in this plant that exceed this)
 - ^{240}Pu concentration between 3 percent and 10 percent
 - Nitric acid concentration at least 4 molar in plutonium solutions
 - Plutonium is the only fissile element (unless other elements receive approval through the Unreviewed Safety Question process, as needed)
 - No visible, separable layer of organic is present in the solution as determined by PFP Analytical Laboratory procedure ZA-519-351, Phase Determination of Samples "
- 2 Confirm with the Building Emergency Director's office (BED) that procedure ZO-100-403, 'Seismic Shutdown Accelerometer Surveillance' will be performed within 24 hours before the test begins and that both accelerometers are working now and are expected to remain operable during the next day's run
 Availability of both accelerometers is a Limiting Condition for Operation (LCO) in the Operational Safety Requirements (OSRs)
- 3 Confirm with the BED's office that there are no planned outages of systems that need to be operable when fissile material is moved
- 4 Confirm that the locks and tags are removed from the pressurized air supply system in Room 188 and from circuit breaker CB 10 in Power Panel PB in Room 262 of the duct level
- 5 Confirm that the pressure differences across the inlet and exhaust HEPA filters are in their acceptable ranges. Notify management or the BED if out of range so that adjustments can be made to get into proper operational ranges
- 6 Confirm that the following equipment work: chiller, heaters, and caustic supply pump. If plant operating conditions allow, the agitator and blowback system should also be checked for proper operation.
 Instructions for testing these components are in the Operability Check section (Section 7.2)
- 7 Confirm that the compressed air oil trap is empty and that the line from there to the collection bottle is unobstructed
- 8 Confirm the availability of certified fissile material handlers and a Nuclear Material Custodian within the PPSL for the timing of the test if using fissile solutions as feed
- 9 Confirm that the building vacuum and pressurized air systems are working and expected to remain working during the run
- 10 Confirm that the balance in the basement section has just been calibrated or will be calibrated before it needs to be used at the end of the run. Calibrations are only good for 24 hours
- 11 When starting the run with fissile solution transfers to Room 188 and then opening the containers in an Airborne Radioactivity Area, the pre-job meeting for the start-up may be held on the day before the run or immediately before starting the run

When reporting to work for the start of a calciner test, repeat Items 1, 2, 3, 4, 5, and 7 for this shift and expected conditions through other shifts until the run is completed. Item 2 must be repeated every 24 hours for the duration of the run (LCO 3.2.3 in Reference 6)

7.2 Operability Check

All the steps in this section were performed for the first operability check in September 1995 except step 13 which is for new equipment added in 1999. Subsequent operability checks might not require repeating every step. The steps may be performed in any order. The engineer in charge of testing will determine/confirm which steps are not needed during the operability check. Step 5 may also be used by itself when reloading product that did not meet storage criteria.

This section may be interrupted, as needed, to obtain craft support for repairs, adjustments, or corrections to equipment and installation. Rigorous compliance with the PFP Lock and Tag program is required for all maintenance activities.

- 1 Turn on power to the control panel at Panel Box AZ. Ensure that all lights, controls, and recorders have power and are operating properly for this stage of operation.
- 2 Program the temperature controllers and recorders, as needed, if not still programmed correctly from previous testing. Programming instructions are in the operating manuals (Ref 7 and 8).
- 3 Open valve V-25 just outside the east side of the glovebox for the building vacuum system and adjust regulator to the desired vacuum level in the calciner. Observe the vacuum indications on PI-3 and PI-4 to confirm that vacuum is available. Getting vacuum into the calciner requires routing through one of the phase disengaging tanks per the valve positioning in step 4, below. Close valve V-25 after adjustments are made.
- 4 Check off-gas routing through both phase disengaging tanks (TKs-3 and 4) by watching for gas bubbles in the scrubber (TK-2). Arrange valve positions as follows for checking each tank.

Disengager in use	Close	Open	Vacuum	Vent
East	V-6 V-8 V-7 V-9	FC-1	V-5	V-4
West	V-6 V-8 V-7 V-9	FC-1	V-4	V-5

Then position V-3 to direct the flow through the desired tank. Open valve V-25. If no bubbles are noticed, reposition V-3 and check other valves' alignment. Observe vacuum gauges PI-3 and PI-4 to confirm that vacuum is available at the glovebox. Once off-gas routings are confirmed, close valve V-25.

- 5 Add the starter bed of PuO_2 to the calciner, as follows:
 - a Start the calciner agitator. Building vacuum may also be used, if available. Product receiver (TK-6) must be in place. The product outlet tube must be as high as it can be raised.
 - b Remove the top part of the pressure relief device to uncover the vent hole.
 - c Insert a funnel into the pressure relief hole.

CRITICALITY

Observe the scrubber solution very carefully for changes in color or clarity while adding PuO_2 into the calciner. Stop the addition and notify management and the Criticality Safety Representative immediately if the scrubber solution appears to be getting darker and/or murkier. These changes indicate that the PuO_2 may be getting into the scrubber through a broken filter element.

- d Carefully pour the PuO_2 from its container(s) into the calciner through the funnel. Tap or shake the funnel gently to assist the powder in flowing down into the calciner chamber. Use a brush or scoopula, if needed, to assist in removing the powder from the original container(s).
 - e When finished loading, brush or shake as much powder as possible from the funnel into the calciner.
 - f Remove the funnel and place the top half of the pressure relief device back in position.
 - g Turn off the agitator and the vacuum (close V 25).
- 6 Add about 0.5 L water to the feed tank (TK 1). Prime the feed pump (P-1) per the operating manual's instructions (Reference 9) with the pump outlet line disconnected from the calciner. Shut off pump P-1 when the first drop of liquid exits the outlet line. Reconnect the feed line to the calciner.

NOTE

The pump stroke frequency may be changed at any time, but the pump stroke length may be changed only when the pump is running.

- 7 Temporarily connect the 30% NaOH fresh scrub solution supply pump (P-2) to a utility outlet and a water supply to prime the 30% NaOH scrubber-chiller feed pump with water per the operating manual's instructions (Reference 10). Open valve V 20 just downstream from P-2 before starting the pump, then prime at 100% flow. Once primed, fill the scrubber-chiller about 2/3 full of water, then shut off P 2 and close valve V-20. Visually check lines and joints for leaks. Insert foot valve into NaOH drum. Reconnect pump P 2 to panel controlled power supply.
- 8 Fill the closed cooling loop between the chiller and scrubber (TK-2) with water. Open valves V 21, V-22, V 23, and V-24, then turn on the chiller per the operating manual's instructions (Ref 9) to begin circulating the cooling water and confirm that the chiller is operating properly. Visually check lines and joints for leaks. Shut off the chiller after confirming acceptable operation, then close V-24 or V-23 in Room 187 (V-24 is preferred).
- 9 Turn on the agitator drive motor and adjust the rotational speed as desired (nominally 60 RPM). Record the rotational speed and necessary dial setting in the laboratory notebook for later reference. Additional speeds and their dial settings may also be determined and recorded, if desired. Turn off the agitator drive system.
- 10 With the control panel Emergency Shutdown switch pushed in (off), open the valve for the compressed air supply (V 2708-23P) and adjust the regulators, if necessary, for the desired line pressure, i.e. 65 psig to glovebox and blow back system and 65 psig to atomizing nozzle. Close compressed air valve V-2708 23P after adjustment is made.
- 11 Turn on the blow-back timing system and adjust, as needed. Nominally 0.5 second pulses every 90 seconds. Turn system off after adjustments are made.

- 12 Confirm that the calciner heater controller is still programmed properly from the previous tests Temperature set point is 1000°C Ramp up rate is 8°C per minute Ramp down rate is 5°C per minute Overtemp alarm at 1150°C Others as directed by laboratory notebook direction Turn on power to the heaters long enough to confirm that (a) the calciner is heating at the proper rate and (b) the increasing temperatures are being recorded on the temperature indicators and recorders Shut off the controller program by depressing the Up Arrow and Down Arrow simultaneously on the controller face
- 13 Request that Maintenance check the correct operation of check valve CV-188 1 on the fresh caustic solution supply line just inside the glovebox at the north end
- 14 Shut off power to the control panel at Power Panel AZ, if desired or necessary

7.3 Start-Up

NOTE

Step 9 may be performed earlier in the start-up including days ahead if desired

Aborted start-ups may be resumed at any point in this section at the discretion of the engineer in charge

- 1 In Room 188, confirm that cooling water valves V 21 and V-22 on the south end of the glovebox are open In Room 187 open valves V-23 and V-24 on the back of the unit, then turn on the chiller and begin circulating cooling water through the scrubber
- 2 Ensure that heat controller and temperature alarm thermocouples are in their proper positions Turn on power to the calciner heaters Check programming for ramp up at 8°C/min or other as directed by laboratory notebook instruction Monitor the heater controller's temperature indication and time to determine the calciner heating rate Record the rate in the laboratory notebook and state whether the engineer in charge determined it is acceptable or not
- 3 Open valve V 25 for the building vacuum system to establish air flow through the calciner and filters Valve FC-1 must be open, but may be partly open at the discretion of the engineer in charge Route the off-gas flow through the desired disengaging tank TK 3 or TK-4) by positioning V-3 for the desired tank, then arranging the off gas valves positioning as follows

Disengager in use	Close	Open	Vacuum	Vent
East	V-6 V 8 V 7 V-9	FC 1	V-5	V 4
West	V-6 V 8 V-7 V-9	FC 1	V-4	V 5

- 4 Open valve V-2708-23P for compressed air flow into the calciner Adjust rotameter FI-1 to indicate 30 scfh (this indication should correspond to an actual 40 scfh after correction for the estimated pressure inside the rotameter) or other rate as specified by laboratory notebook instruction Set rotameter FI 2 to about 0.5 scfh or other flow rate recorded in the laboratory notebook

- 5 Turn on the agitator drive motor. Adjust its speed to 60 RPM or other as directed by laboratory notebook instruction.
- 6 Turn on the filter blow-back system.
- 7 Push in the button shaped Emergency Shutdown switch and confirm that the agitator stops, the pressurized air flow stops, and the heaters shut off. If any of those systems remain operating, abort the test and shut down per Section 7.5.
- 8 If the pressurized air, heaters, and agitator stopped as needed, then turn the speed control knob for the agitator to "0" then pull the Emergency Shutdown switch back out to resume start-up. Turn the speed control knob for the agitator slowly back to its original position and confirm that the air, agitator blowback system, and heaters are running (their Start switches may need pushed in again).

CRITICALITY

Only one feed container, empty or full, is allowed inside the glovebox at any time. The emptied container must be removed or made into a non-container before the next feed container may be brought into the glovebox.

- 9 As the calciner heats toward operating temperature, introduce feed solution as required into the glovebox per ZP 100-009 "Change PPSL Glovebox Gloves and Bags" or ZP-100 003 "Use PPSL Airlocks" and thence, into the feed tank. Check the feed solution for a second, separate phase, shut down if one is noticed. Enter on inventory posting sheet and into notebook. Remove each emptied feed container per ZP 100-001, "Seal Out PPSL Gloveboxes" before bringing another feed container into the glovebox.
- 10 After the temperature indicator in the heater controller indicates the desired furnace operating temperature 1000°C or other as directed by laboratory notebook instruction, increase the compressed air flow rate at rotameter FI-1 to an indicated 45 scfh (estimated actual 60 scfh) or other as directed by laboratory notebook instruction.
- 11 Turn on caustic solution supply pump P-2 and begin pumping 30% NaOH solution into the scrubber at a rate estimated to yield an effluent ca. 1.5M in excess hydroxide as directed by laboratory notebook instruction from prior makeup calculations or previous experience.
- 12 Open the feed tank outlet valve (V-2) and lower the clean-out rod at the feed injection port area. Then, turn on the feed pump (P-1) to begin pumping feed solution into the calciner at 4.0 L/hr or other as directed by laboratory notebook instruction. Observe rotameter FI-1 in the middle level of the glovebox to confirm that air is still flowing properly into the calciner and that feed solution is NOT flowing backward through the rotameter.
- 13 Confirm that valve V-1 between the calciner and product collection pot (TK-6) is open.
- 14 Confirm that feed solution is flowing into the calciner by observing the following:
 - off gas filter dP gauge DPI-1 for an increase in pressure drop due to the extra gases generated with each feed pulse
 - the feed line from the pump to the injection port area for a change in color from clear to brown (does not occur if using just acid solution for feed)

7.4 Steady-State Operation

- 1 Periodically check the solids content of the product collector (TK-6)
- 2 When the collector (TK-6) is nearly full, empty the collector as follows

CRITICALITY

Only two 0.5 L containers may be present empty or full in the calciner basement at any time

- a Tare weigh a slip lid can. Assign a number to this can. record the can number and tare weight in the laboratory notebook or other location determined by the test engineer in accordance with any applicable classification restrictions
- b Close valve V-1 between the calciner and product collection pot (TK-6)
- c Carefully undo the clamps holding the collection pot in place making certain that the pot does not fall
- d Very carefully pour the collected powder from the collection pot into the tared slip-lid can. Use brushes or other instruments, if needed to minimize dusting
- e Reattach the collection pot and open valve V 1 between it and the calciner
- f Tare weigh a 10 mL plastic graduated cylinder. Introduce 5 to 7 mLs of product PuO_2 into the cylinder. Tap down a few times. Measure and record volume and weight. Return sample to slip-lid can
- g Weigh the loaded slip-lid can. Record the gross weight in the laboratory notebook. Calculate the net weight of powder and record this amount in the notebook. [Note: The gross weight and net weight may be the same if the balance has an automatic tare feature]
- h Tare weigh a numbered sample bottle. Remove ten grams of powder from the slip lid can and transfer it into the sample bottle. Reweigh the sample bottle to obtain the sample weight. Attach the sample bottle cap and set the sample aside for later submission to PFP Analytical Services for Loss on Ignition and tap density analyses

CRITICALITY

Containers of fissile material must be kept at least 10 inches from each other and from fixed equipment containing fissile material except when transferring contents from one container to another

- i Place the lid on the slip lid can and seal with tape. Sealed cans must be kept just outside the open corners of the triangular plate beneath the product collector or further apart
- j Calculate product PuO_2 tap density. Note and record. The density may not exceed 5.5 g Pu/cm^3 (i.e. 6.24 g PuO_2/cm^3) bulk density. Should the measured bulk density exceed 6.23 g/ cm^3 , stop operation, notify the PFP Criticality Representative
- k Label the slip-lid can per the requirements of ZP-100-004 "Label Nuclear Material"
- l Bag the slip-lid can out of the glovebox per ZP-100-001, "Seal Out PPSL Gloveboxes"

CAUTION

If the bagged slip-lid can will not fit into the first seam-sealable can or if anything else goes wrong with Step m (below) the assembly may be sealed back into the glovebox per ZP-100-009 "Change PPSL Glovebox Gloves and Bags" to repeat Step l (above). An outer seam-sealed can may be opened first outside the glovebox to reveal the inner seam-sealed can, but the inner seam-sealed can must be opened inside the glovebox to prevent cutting the seal out bag with the can opener. Removing both the top and bottom surfaces of the inner can allows easier removal of the bagged slip lid can and also allows flattening the can into a non-container.

- m Seal the bagged slip-lid can into two outer cans per ZP-100-015, "Operate PPSL Can Sealer and Inspect Double Seam on Seam-Sealed Cans"
 - n Move the seam-sealed can to Room 191 per ZSP 100-002, "Move Fissile Material"
 - o Label the can per ZP-100-008 "Apply Pressure-Sensitive Seals in PPSL"
 - p Seal out the samples and package for transport to PFP Analytical Services
 - q Move the can to its storage location and store per ZSP-100-002 "Move Fissile Material"
 - r Notify the Building Emergency Director (BED) that one or more cans of calciner product require 4-hour surveillance per Operating Specification Document OSD-Z-184 00013, *Operating Specifications for Plutonium Finishing Plant*, Section 13.2, "Solid SNM Items Packaged After January 1 1998" Variable D (Weight Percent Volatiles) Specification 3
 - s When present PPSL personnel shall perform the monitoring required in Step q (above) per ZP-100-006 "Monitor for Pressurized or Suspect Cans in PPSL"
 - t When Analytical Services returns the Loss On Ignition result for the can(s), add the LOI value(s) to the seals per ZP 100-008, "Apply Pressure-Sensitive Seals in PPSL"
 - u If the LOI value is acceptable for storage then notify management and the BED that the pressurized can surveillances may end
 - v If the LOI is unacceptable for storage, then notify management that the can must be returned to Glovebox 188.1 for reheating unless another alternate treatment method is available and preferable. Notify the BED when the can is removed from its temporary storage location to end surveillances
- 3 Monitor the time remaining in the Dwell portion of the temperature controller program. This indication tells you the number of minutes remaining at steady state operation. The controller will automatically begin to cool at the programmed ramp-down rate when the Dwell time reaches 0.0 minutes. Depress the Run/Hold button any time during the Dwell step to hold the heaters at steady-state operation for as long as necessary. The Dwell step will resume where it stopped when the Run/Hold button is depressed again.

CRITICALITY

The scrubber (TK 2) and the disengagers (TK 3 and TK 4) are critically safe by geometry for plutonium slurries but ALARA principals make it necessary to limit plutonium contents in solutions going to the waste tanks.

Under no circumstances are gross quantities, i.e. approaching the 400g Pu mass limit of the 241-Z receiver tanks to be introduced into the D-8 drain system.

If marked turbidity is noticed shut down feed and scrub solution pumps (P 1 and P 2), pressurized air and blowback air (close V 2708 23P), and off-gas vacuum (close V-25) immediately. Turn off heater power subsequently. Notify management and the CSR immediately after shutting down.

- 4 Periodically examine the contents of the scrubber (TK-2) in the lower annular downcomer and the quiescent chamber beside the gas distributor and/or the scrub liquor effluent receiver tanks for turbidity. Marked turbidity indicates a failed off-gas filter. Do not mistake finely dispersed gas bubbles for turbidity. Scrub solutions can be slightly turbid with a grayish material, presumably sodium carbonate.
- 5 Observe the feed tank volumes and times to confirm the feed flow rate.

CRITICALITY

Be certain to check all aspects of the CPS posting before introducing additional feed solution into the glovebox.

- 6 Periodically add more feed solution to the feed tank (TK 2) per ZP-100-009 "Change PPSL Glovebox Gloves and Bags," or ZP-100-003 "Use PPSL Airlocks" as needed. Note volumes and concentrations. Enter onto inventory posting sheet and into laboratory notebook. Project necessary scrub solution flow rate in notebook calculation. Change scrub solution feed rate as appropriate.

CRITICALITY

Do not drain phase disengager (TK-3 or TK-4) if it exhibits marked turbidity. Instead, shut down feed and scrubber solutions (P-1 and P-2), pressurized air and blowback air (close V 2708-23P), and off gas vacuum (close V-25) immediately. Turn off heater power subsequently. Notify management and the CSR immediately after shutting down.

- 7 Periodically check the scrub solution disengagers system liquid levels (TK 3 and TK-4). Make certain that the vacuum trap (TK 5) is operating acceptably. Adjust valving, as needed, to switch between disengagers during operation (see Step 4 in Section 7.3 "Start-Up," for valve arrangements). Adjust manual valve FC-1 as needed, per the direction of the engineer in charge. Drain the disengager not in use as needed, per the following instructions:
 - a Open the vacuum valve (first)(V-4 or V-5) then the drain valve (V 8 or V-9) on the tank to be sampled. Sparge the contents of the tank for at least two minutes to mix the contents for obtaining a representative sample.
 - b Close the drain valve (V 8 or V 9).
 - c Set appropriate valve V-4 or V 5 to Vent.
 - d Drain a sample of the solution from the "full" disengager into a numbered 22 mL sample bottle through the tank's sample valve (V-8 or V-9).
 - e Close the sample valve.
 - f Seal out the sample(s) and submit it/them to PFP Analytical Services.
 - g After receiving sample results and approval of the 241 Z Cognizant Engineer and the PFP on-duty Surveillance Team, notify the Building Emergency Director that one (or both) disengaging tank is ready to drain to the D-8 system. When approved by the BED, open the valve to the radioactive drain (V-6 or V 7) and set the vac/vent valve (V-4 or V 5) to Vent.

- h When the tank is empty close the drain valve
- 8 Perform frequent checks of air rotameter FI-1 in the middle level of the glovebox and the feed pump's bypass loop. Confirm that air is flowing properly into the calciner and that solution is not flowing backward through rotameter FI-1. Loss of air flow, liquid flowing through the bypass loop, or backflow of solution through the rotameter indicates probable feed line pluggage that can be cleared as follows

CAUTION

If a significant quantity of feed solution is known or suspected to have accumulated in the pressurized air line(s), shut down and contact Maintenance for disassembly of the feed and/or air lines. The steps below are for use only when the loss of air flow is noticed quickly enough to prevent a large amount of feed from entering the calciner rapidly when the plug is cleared

- a Turn off the agitator
- b Shut off the feed pump (P-1). Relieve the feed pump pressure and allow any feed in the feed and air lines to blow back into the feed tank by pulling the priming knobs on the feed pump outlet assembly. Close these knobs before the feed line is totally empty.
- c Turn off the air supply to rotameter FI 1 by closing V-2708-23P. In the top level of the glovebox, relieve the pressure in the accumulator tank(TK-7) by opening drain valve V-11.
- d Loosen the gland nut at the base of the feed tube assembly to free the clean-out rod.
- e Push the clean out rod slowly and carefully upward all the way to remove the suspected pluggage. If the rod will not move all the way up, shut down and contact Maintenance for disassembly to correct the problem.
- f Return the clean-out rod to its original position and tighten the gland nut loosened in step d.
- g Turn the pressurized air supply back on by opening V-2708 23P. Confirm that air is flowing properly through rotameter FI 1. If air does not flow through rotameter, shut down and contact Maintenance.
- h Turn on the agitator.
- i Turn on the feed pump (P-1). If needed, reprime the pump per the operating manual instructions.

7.5 Shut-Down

- 1 As the feed tank (TK-1) nears empty with the last planned feed for the run, load in a 0.5-L polyjar of dilute acid through the sphincter port (not necessary when using just acid solution as feed). Solution of about 1.5 M nitric acid is preferred to guarantee that polymer can not be formed, but more dilute solutions are acceptable. Water is also acceptable.
- 2 When the feed solution in the feed tank disappears down into the transfer line going to the feed pump, add the dilute acid from the 0.5-L polyjar to the feed tank.

- 3 When the last of the flush solution disappears from the feed tank into the transfer line turn off feed pump P-1 and close valve V 2 between the pump and feed tank Raise the clean-out rod at the feed injection port to its uppermost position
- 4 Allow the temperature controller to finish its programmed Dwell portion of the operating program and it automatically begins to cool
- 5 Before leaving the calciner unattended, shut off the agitator scrub solution pump (P-2), compressed air (close V-2708 23P), off gas blowback system and vacuum(V-25) Be sure the compressed air and blowback system are shut off BEFORE shutting off the vacuum Close the ball valve downstream of the caustic pump (V-25) after shutting that pump off The master power switch in Power Panel AZ may also be turned off if desired
- 6 Turn off the power to the calciner heaters Then push in the button-shaped Emergency Shutdown switch
- 7 Wait about 20 minutes after shutting off the feed pump to allow the bed to stop decomposing and producing NO_x then remove the cap on the pressure relief device If fumes exit the relief port replace the cap and wait longer before leaving the cap off
- 8 Any product needing to be canned and removed should be allowed to cool to <50°C before handling The canning and load-out are done per the instructions in Section 7 4, Step 2
- 9 After leaving Room 188, enter Room 187 and shut off the chiller Close valve V-24 on the back of the unit to prevent contaminated solution from entering the chiller reservoir in case of a leak This step may also be performed by another worker already out of Room 188 directly after Step 5 above
- 10 If the shut down will be long-term (no future runs maintenance or other calciner activities planned), then close pressurized air supply valve V 2707-23P (gate valve near ceiling at north end of glovebox) and request the on-duty Surveillance Team to install a Caution tag on it Also request the on-duty Surveillance Team to open and install a Caution tag on breaker CB 10 in Panel PB in Room 262 These steps may also be implemented at the discretion of the responsible engineer at any time

7 6 Emergency Conditions Responses

1 Criticality Howler

If possible without pausing or stopping, shut off power at the button shaped Emergency Shutdown switch on the control panel if passing by the panel Run from the building immediately per normal criticality siren response as required by ZCR-008

2 Continuous Air Monitor

If possible without pausing or stopping shut off power at the button-shaped Emergency Shutdown switch on the control panel, if passing by the panel Leave the room and contact the Building Emergency Director and Radiological Control Technicians (RCT)as required by ZCR-001

3 Evacuation Siren

Shut off power at the button shaped Emergency Shutdown switch on the control panel Shut off any other powered devices by depressing their red lights on the control panel Shut off the compressed air supply by closing V-2708 23P (first) and the vacuum supply valve V-25 (second) Crack open the cap on the pressure

relief device at the calciner top Evacuate the room and building per normal evacuation siren response as required by ZCR 009

4 Fire Alarm

Shut off power at the button-shaped Emergency Shutdown switch on the control panel Shut off any other powered devices by depressing their red lights on the control panel Shut off the compressed air supply by closing V-2708-23P (first) and vacuum supply valve V-25 (second) Crack open the cap on the pressure relief device at the calciner top Evacuate the room and building per ZCR 005

5 Take Cover Siren

No response is required Continue operating normally unless instructed to shut down by management

6 Orderly Exit of Zone III

Shut down normally per Section 7.5 Notify the building Emergency Director that the shut-down and exit will require about half an hour If directed to leave sooner use the response for an evacuation siren but without leaving the building

7 Fissile Material Movement Bans/Restrictions

Shut off power at the button-shaped Emergency Shutdown switch on the control panel Shut off any other powered devices by depressing their red lights on the control panel Shut off the compressed air supply by closing valve V-2708-23P (first) and the vacuum supply valve V-25 (second) Shut off the caustic pump (P 2) and close the ball valve on the caustic line (V 20) After leaving the room, shut off the chiller in Room 187 and close valve V-24 on the back of that unit

8 Lock-Downs

No response is required unless specifically directed by Patrol or management If directed to leave respond as per the evacuation siren instructions but without leaving the building

9 Seismic Events

REMAIN CALM If possible get to a doorway and brace yourself until the tremor stops All electrical power to the glovebox and the pressurized air to the glovebox will stop automatically via the seismic shutdown system If going by the control panel during or after the tremor, push in the emergency shutdown switch, if possible

After the tremor stops evacuate quickly, but carefully, to the staging area and follow the BED's instructions Assist others as needed and able to get everyone out of the building Do not stop to survey anywhere on the way out

10 Breached Gloves

Push in the button-shaped Emergency Shutdown switch and shut off the pressurized air by closing V-2708 23P Then respond per ZCR-012

8 QUALITY ASSURANCE

These tests are being performed to the requirements for prototype equipment as detailed in *Development Control Requirements* (B&W Hanford Company facility specific procedure FSP-442) There are no requirements for independent witnessing or review of tests nor are there any specific standards to be met Equipment will not be calibrated to NIST-traceable accuracy

9 ORGANIZATION AND FUNCTION RESPONSIBILITIES

9 1 Plutonium Process Support Laboratories

The PPSL staff prepares all test plans and procedures, performs all tests and documents results from testing The PPSL Cognizant Engineer also reviews and approves all designs and procurement for equipment and its installation The PPSL technical staff also prepares formal presentations based on the test results

9 2 PFP Engineering

Engineering prepares all fabrication and installation designs and relevant bills of material Engineering also reviews and approves this test plan Documentation and presentation of designs is also performed Engineering also observes the tests as needed

9 3 PFP Maintenance Crafts

Maintenance crafts from various organizational teams review designs fabricate and install the equipment for use and may observe the tests

9 4 PFP Surveillance Teams

The PFP Surveillance Teams perform the required surveillances for the seismic shutdown system approve transfers of spent scrub solution to the building radioactive drain system and coordinate building utilities, as needed

9 5 Solid Waste Operations

Solid Waste Operations assists with the disposal of all effluents and wastes

9 6 Safety

The PFP CSR reviews and approves equipment designs for criticality safety, assists with the CSER and writes the CPS and associated posting(s) The CSR also reviews and approves the test plan for criticality safety concerns The CSR inspects the PPSL area periodically to confirm compliance with all applicable CPS postings The CSR also observes the tests as desired

The PFP Nuclear Safety representative reviews and approves this test plan and periodically performs surveillances to ensure compliance with nuclear safety requirements, including criticality safety The nuclear safety representative also observes the tests as desired

The PFP Industrial Safety Representative reviews and approves this test plan and performs periodic surveillances to ensure compliance with industrial safety rules The industrial safety representative also observes the tests, as desired

9 7 Quality

Quality personnel review and approve designs and this test plan. Test observations may be performed as desired.

9 8 PFP Environmental

The PFP Environmental Team reviews records of effluent solutions and assists in their disposal. PFP Environmental Engineering also reviews and approves this test plan. Test observance may be performed, as desired.

9 9 PFP Security

The PFP Security Representative reviews and approves the material requirements and associated safeguards for special nuclear material (SNM) protection. Test observance may be performed, as desired.

9 10 Department of Energy

Department of Energy personnel or their representatives review the test plan. Test observation may be performed, as desired.

10 SCHEDULE

These tests are expected to begin on July 1, 1999. Tests are anticipated to continue until all test objectives are met. The duration of testing will not be predicted due to the uncertainties in the numbers of solutions to be tested and any required maintenance or design changes.

11 REPORTS

A final test report will be issued after completion of these tests. It will be issued as a supporting document, external publication, or speech/article in accordance with *Supporting Document Requirements* (BWHC facility specific procedure FSP-439). Progress of the testing will also be reported weekly in the PPSL Weekly Report.

12 REFERENCES

- 1 Fluor Daniel Northwest, Inc. HNF-4224 Revision 0, *Laboratory Prototype Calciner Addendum to the Plutonium Finishing Plant Facility Safety Analysis Report*, Richland, WA, June 1999.
- 2 Shapley, J. E. HNF-SD-CP SAR-021, Rev. 0-J, *Plutonium Finishing Plant Final Safety Analysis Report*, Fluor Daniel Hanford Company, Richland, Wash., September, 1995.
- 3 Compton, J. A., WHC SD-CP-TA-008 Rev. 0, *Comparison of Deliverable and Exhaustible Pressurized Air Flow Rates in Laboratory Gloveboxes*, Westinghouse Hanford Company, Richland, WA, August 1994.
- 4 B&W Hanford Company, *Plutonium Finishing Plant Administrative Procedures*, Administrative Procedure ZAP 100 010, PFP Chemical Hygiene Plan, Richland, Wash., September, 1998.
- 5 Keck, R. D., HNF-SD-CP-SDD-004, *Definition and Means of Maintaining the Supply Ventilation System Seismic Shutdown Portion of the PFP Safety Envelope*, B&W Hanford Company, Richland, WA, (to be issued).

- 6 B&W Hanford Company WHC-SD-CP OSR-010, Rev 0-J, *Plutonium Finishing Plant Operational Safety Requirements*, Richland Wash , June, 1999
- 7 Eurotherm Corporation Bulletin 8180P-8 Eurotherm 818 Controller/Programmer Operating Instructions, Eurotherm Corporation Reston, VA
- 8 ABB Kent-Taylor, Inc Bulletin PB PR100-94 2 "Commander™ PR100 Advanced Process Recorder " ABB Kent Taylor, Inc Rochester, NY, 1994
- 9 LMI Corporation, Liquid Metronics Division Catalog Section 6 0 "Electronic Metering Pumps Installation Maintenance, Troubleshooting " LMI Milton-Roy Acton, MA, July 1994
- 10 Neslab Instruments Inc , "CTF 300 Recirculating Chiller, Instruction and Operation Manual " Neslab Instruments Inc , Newington NH September 9, 1994

13 DATA SHEETS

Data will be recorded in Controlled Laboratory Notebook WHC-N-967-1, with recorder traces or any other mechanically produced data stored separately as noted in that notebook Those data items will be identified on the PPSL RIDS plan as record material Any classified data may be recorded and stored separately